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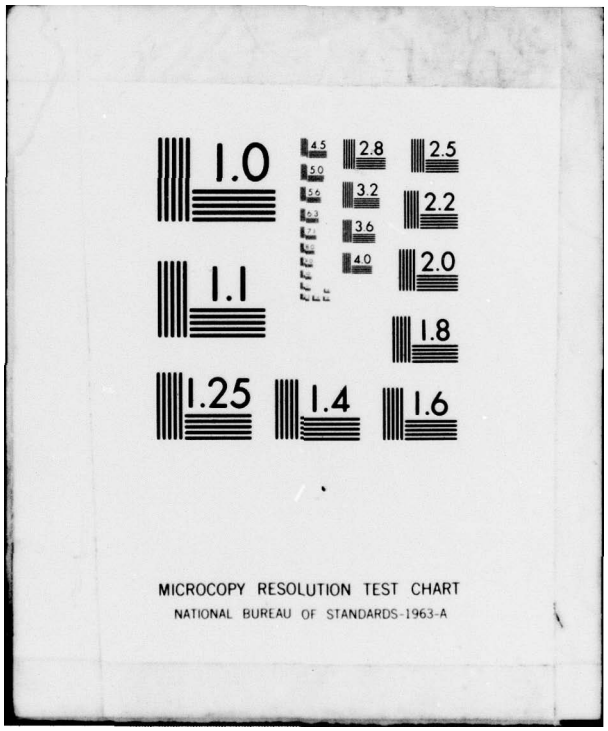
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The authors have found a new technique for the synthesis of metastable compounds of well defined composition: namely, ion implantation of a selected element into the desired crystal structure. Starting with a substrate material of A-15 $Nb_3Al_{0.9}Si_{0.1}$, two basic approaches were tried towards the formation of A-15 Nb_3Si by Si implantation: 1) direct replacement of the Al by Si, and 2) implantation into a surface layer depleted of Al. This latter approach proved to be the most successful. It consisted of removing the Al by a → (over)

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20. Abstract

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TECHNICAL REPORT No. 2

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"ON THE SYNTHESIS OF METASTABLE A-15 "Nb_3Si" BY ION IMPLANTATION AND ON ITS SUPERCONDUCTING TRANSITION TEMPERATURE"

by

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November, 1979

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ABSTRACT

The authors have found a new technique for the synthesis of metastable compounds of well defined composition: namely, ion implantation of a selected element into the desired crystal structure[1]. Starting with a substrate material of A-15 $Nb_3Al_{0.9}Si_{0.1}$, two basic approaches were tried towards the formation of A-15 Nb_3Si by Si implantation: 1) direct replacement of the Al by Si, and, 2) implantation into a surface layer depleted of Al. This latter approach proved to be the most successful. It consisted of removing the Al by a diffusion anneal and replacing the Al deficiency by sequential Si implantations. Upon subsequent heat treatment a surface layer of A-15 $Nb_3Al_{0.2}Si_{0.8}$ was produced. Details of the experimental procedure and a discussion of the superconducting transition temperature measurements of the implanted surfaces are presented.

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INTRODUCTION

The authors have recently reported on a novel application of ion implantation for the synthesis of metastable or unstable compounds of well defined composition[1]. They applied the new technique to the formation of metastable A-15 "Nb_3Si". This compound is of interest because of the predictions that it would have a transition temperature for superconductivity greater than 25 K. The authors have also reported on the superconducting transition temperature measurements of the implanted surfaces[2]. The highest T_c they obtained was 5.6 K. This paper will present the details of the experimental procedure and a discussion of the results.

EXPERIMENTAL PROCEDURE

The basic process involved the incorporation of Si into an A-15 substrate by ion implantation. The substrate material chosen was $Nb_3Al_{0.9}Si_{0.1}$ for the following reasons. The addition of 3% Si to Nb_3Al increases the T_c from 18.7 to 19.2 K[3]; this is the highest concentration of Si that has been incorporated into a single phase A-15 lattice by standard metallurgical techniques. Si may stabilize the A-15 structure so that the σ phase does not precipitate out during heat treatments as it does for Nb_3Al[3]. The bulk concentration of Si can be used to determine the surface concentration of Si as determined from ion microprobe analysis.

The Al and Si are of similar atomic mass which may favor direct Al-Si replacement. The lattice parameter of $Nb_3Al_{0.9}Si_{0.1}$ is 5.174 Å [3] and that of A-15 Nb_3Si is predicted to be 5.09 Å [4] ; these are within 2% of each other which is well within the range for epitaxial recrystallization.

Samples were prepared by arc melting compacted powders. High purity powders (Nb, -325 mesh, 99.8% metallic purity; Al, -325 mesh, 99.8% metallic purity, 99% total purity; Si, -100 mesh, 99.9 +% metallic purity), were weighed, mixed with a mortar and pestle and compacted in a stainless steel dye at 36000 psi. Pellets weighing ~10 grams were then arc melted on a water-cooled copper hearth in a titanium gettered argon atmosphere, using a nonconsumable tungsten electrode. Al was lost during this process and compensatory additions were made to the initial powder mixtures. The samples were given a homogenizing anneal at 1840°C for 2 hours. They were then given ordering anneals in a dynamic vacuum furnace at 10^{-6} torr. Anneals of 48 hours at 750°C and 100 hours at 700°C gave the highest T_c's (19.2 K) The crystal structures were determined from X-ray diffraction patterns. Single-phase A-15 material was obtained if the correct amount of Al was evaporated during the arc melting, and if the samples were cooled sufficiently rapidly from the 1840°C heat treatment. However, this material was extremely brittle and often cracked upon cooling from 1840°C; it also proved difficult to slice. Since we needed smooth clean slices for the implantation work, we decided to use samples with a small amount of σ phase; these were much less brittle and easier to work with. The slicing of the ingots was done with a diamond cutter or a spark slicer.

The spark planer was used to smooth and flatten the slices, readying them for the final stages of polishing. The slices were mounted on brass "parallel" polishing holder with jeweler's wax and polished in the following way: 320 and 600 grit SiC paper, dry, by hand on a microflat; 1 diamond paste on silk cloth, lubricated with diamond polishing compound, with a polishing wheel rotating slowly (~1 minute for small samples and 3 minutes for large samples); 1/4 μ diamond paste, no more than 30 seconds; 0.1 and 0.03 μ alumina, very briefly. Finally, the slices were given a light etch for 10 seconds in 2HF, 18 H NO_3 and 5 lactic.

Four point probe resistive methods were used to measure the superconductivity transition temperatures. When it was determined that the implanted layer had a lower T_c than that of the substrate, the following probe configuration was used: current and voltage probes were placed on the front and back of the wafer in such a way that the current was passing perpendicular to the surface. The substrate material is ~0.2 mm. thick and the implanted layer should be ~1/2000 of that of the substrate. A fairly large current can be used since it penetrates a cross sec onal area of implanted material approximately 1 mm. square. Typical currents used were 10 to 20 mA. For our particular case, where normal resistivities are of the order of 10^{-6} ohm-meters and the implanted layer thickness is ca. 10^{-7} meters, a voltage drop of 10 nanovolts is typical. This can be detected with a nanovoltmeter as long as great care is taken to reduce the noise level to a minimum. This method was checked experimentally by evaporating thin (1000 Å or 10^{-7} m.)

layers of In and Pb onto Nb discs. The resistive transitions of In and Pb were both clearly seen.

In some cases, before implanting with Si, the concentration of Al near the surface was reduced by annealing in vacuum. The vapor pressure of Al in $Nb_3Al_{0.9}Si_{0.1}$ at 1050°C is sufficiently high that the concentration of Al at the surface is negligible, thus driving the diffusion. The diffusion profile can be predicted by using the following diffusion equation of composition as a function of distance and time:

$$C(x,t) = C_o \text{ erf } [\frac{x}{2\sqrt{Dt}}]$$

The diffusion coefficient D of Al in Nb_3Al is known to be $2.4\ e^{-86000/RT}$ [5]. Samples were wrapped in Nb foil and heat treated in a dynamic vacuum furnace at 10^{-6} torr.

It is possible to predict fairly accurately a mean projected range R_p and a range straggling ΔR_p for the implanted ions. For Si^+ ions into Nb_3Al and into Nb substrates R_p and ΔR_p at different implantation energies are given in Table I[6].

Two ion implantation machines at the M.I.T. Lincoln Laboratories were used[7]. Care was taken to assure that the Si_{28} beam was not contaminated with N_2 or Co[1]. Very high beam currents (~100 μA) were generally used, permitting doses of 5×10^{16} ions/cm^2 to be achieved in a few hours. The intense ion doses caused the samples to heat up during implantation; their average temperature was estimated to be between 100 and 200°C. Prior to and after implantation the samples were kept in evacuated dessicator jars in order to maintain as clean surfaces as possible.

Composition as a function of depth was determined for Nb, Al and Si from the Ion Microprobe Facility of the Materials Science Center (M.I.T.). Quantitative compositional analysis is not obtainable directly; however, it can be inferred to within several percent from the known composition of the bulk, as will be described shortly. The basic process of the Ion Microprobe is to bombard the sample with a beam of ions, and erode the surface at controllable rates. The atoms are sputtered off the surface and analyzed with a mass spectrometer. The instrument can profile composition with a depth resolution of the order of 10 Å, from the surface to a depth of several microns. It is sensitive to all elements with detection limits of the order of 1-10 parts per million. Typical sputtering rates were 1 Å per second, and the samples were analyzed to a depth of ~5000 Å. The crater size was around 1 to 10 μ across. Analysis of ion microprobe data is not straightforward. Composition as a function of depth was determined for Nb, Al and Si by assuming that there was a linear relation between counting intensity and composition. Since the composition of the bulk interior is known, the surface composition can be determined by comparing surface and bulk counting rates. The validity of the above assumption was checked in the following way. Samples were implanted with Si to compositions varying from 4 to 20 At%, as calculated from ion implantation theory. The surface compositions were then determined from ion microprobe analysis using the linear relationship assumed above; these measured compositions agreed with those predicted

from ion implantation data. By averaging sputtering runs, it is believed that composition as a function of depth could be determined to within a few percent. To convert sputtering time to distance, the depth of the crater produced by the ion beam was estimated using a Micro Michelson dual beam interferometer.

It is important that we know the structure of the surface layer before and after implantation, and before, during and after annealing. Reflection Electron Diffraction (R.E.D.) seems to be the best technique for our purposes. R.E.D. makes use of electrons in the 10 to 100 KeV range, corresponding to wavelengths of 0.12 to 0.04 Å. The electron beam strikes the surface at nearly grazing angles of incidence so that only a thin surface layer is penetrated, typically in the 50 to 200 Å range. It is not possible to obtain accurate lattice constants from R.E.D. It is generally considered that $\sim$1% is the best that can be done. However, it is certainly possible to identify structures, to distinguish between them, and to detect structural changes. A special hot stage was designed such that heat treatments up to 1000°C could be done directly in the electron microscope while taking R.E.D. photos. The R.E.D. apparatus is equipped with heater holders that go up to 400°C. One of these was modified by inserting a 10 mil niobium coil in such a way that temperatures of 1000°C could be reached. Samples were attached to the coil with Sauereisen adhesive cement. A fine thermocouple positioned on the sample was used to calibrate its temperature in terms of the settings on the R.E.D. heater control unit.

RESULTS

The first approach toward synthesis of A-15 Nb_3Si was to implant Si directly into an A-15 $Nb_3Al_{0.9}Si_{0.1}$ substrate with the hope of direct Si-Al replacement. There is a good likelihood that the Si ion will come to rest in a substituional site. It is also more probable that the Si will knock out an Al which is of similar atomic mass than a Nb which is three times heavier. However, one has to provide a means for the extra Al to escape the lattice. If low energy ions are used, they remain sufficiently close to the surface that the interstitial aluminums may have a good chance of migrating out of the material via the numerous defects that are created during implantation. For this reason an energy of 50 KeV was chosen, corresponding to a depth range R_p of 357 Å and a range straggling ΔR_p of 200 Å (see Table I). Four implantation doses were chosen of 10^{15}, 3×10^{15}, 10^{16} and 3×10^{16} ions/cm^2, corresponding to concentration $\bar{n}$ at R_p of 0.3%, 1.0%, 3.5% and 10.5%. These should be added to the initial 3.0% Si concentration to obtain the maximum Si concentrations in the alloys of 3.3%, 4.0%, 6.5% and 13.5%. The samples were subjected to various recrystallization and ordering anneals, but it was impossible to detect a superconducting transition for the implanted layer. We concluded that implanting directly into A-15 $Nb_3Al_{0.9}Si_{0.1}$ was producing a surface layer that was considerably off stoichiometry and that had an excess of "B" atoms. This could greatly lower its T_c perhaps to the point that it was not superconducting at all. We, therefore, decided to try a second approach towards synthesis of A-15 "Nb_3Si."

The second approach involved implantation into a surface layer depleted of Al. By annealing the samples in vacuum, the Al is made to diffuse out of the surface. The Al deficiency is then replaced with Si by sequential Si implantations at different doses and energies. In this way it is possible to maintain the (Al + Si) concentration equal to the desirable stoichiometric composition of 25 At%, necessary for high T_c materials[1]. The concentration of Si was increased from 3 At% in the bulk to 20 At% at the surface. The surface structures of the samples were determined from photographs of the relfection electron diffraction rings. The original surface was A-15 with fine continuous diffraction rings. An R.E.D. photo taken at 80 KeV is shown in Figure 1. The ring identifications and Miller indices are given in Figure 2. During the Al depletion, the A-15 surface collapsed into bcc Nb. After implantation the surface structure remained bcc Nb but the diffraction rings were broader and more diffuse, indicating a significant amount of disorder had been introduced. An R.E.D. photo taken at 80 KeV and the corresponding ring identifications are shown in Figure 3 and Figure 4.

The samples were then subjected to a series of recrystallization anneals. The structure transformed from bcc Nb to A-15 in less than an hour at 800°C.[1] The usually observed structure for Nb_3Si is the Ti_3P structure[8]. A comparison of the diffraction lines for the A-15 and Ti_3P structures is shown in Figure 5. They are very different, there being many more lines for the Ti_3P structure than for the A-15. We, therefore, believe there is no ambiguity in distinguishing between the two. It thus appears

from the compositional and structural analyses that the implanted layer has recrystallized into A-15 $Nb_3Al_{0.2}Si_{0.8}$.

In an attempt to optimize the superconducting properties of the implanted layer, the following variables were controlled: stoichiometry, recrystallization anneals and ordering anneals. A series of samples was made up in which the (Al + Si) concentration was varied in approximately one percent steps[2].

Samples were then subjected to recrystallization anneals of 800, 850, 900 and 980°C for 1 hour. The implanted layer became A-15 at 800°C and remained at least as high as 980°C. The ordering anneals chosen ranged from ~500 to 700°C for ~20 to 100 hours. These were chosen because the optimum ordering anneal for $Nb_3Al_{0.9}Si_{0.1}$ is 650°C for 100 hours[3]. Transition temperatures of the implanted layers were measured and the maximum value obtained was 5.6 K^2. A typical transition curve is shown in Figure 6 and can be compared to a curve for a nonimplanted sample. The superconducting voltage drop was ~10 nano V. This is the right order of magnitude as predicted above. Critical field measurements of T_c as a function of magnetic field (up to 150 K gauss) were done. Implanted and unimplanted samples were compared but no difference was observed. A typical T_c versus magnetic field curve is shown in Figure 7.

DISCUSSION

There are several interesting aspects to the previous results.

1. Extremely high implantation doses were used and it is to be expected that a great many defects were introduced during implan-

tation. The fact that we were able to achieve the predicted dopant profiles indicates that enhanced diffusion due to defects was not a problem. These high doses should be compared to typical doses used in semiconductor research where ions are implanted in fractions of a percent. Most of the dopant profile theories have been tested on semiconductor research. The fact that these theories can still be applied for extremely high doses is indeed encouraging. It should be noted that we were helped in this case by the fact that we were implanting a light ion with a low sputtering yield S. (S = # of atoms ejected/# of incident ions.) For heavier ions the sputtering yield can increase to the point where only a few percent of the incident ions actually enter the lattice.

2. There was no large redistribution of atoms during the recrystallization anneals that took place at reasonably high temperatures. This is also encouraging. This implies that we can put atoms where we want them and keep them there. Indeed, we have been able to increase the Si concentration in the A-15 lattice from 3 to 20 At%.

3. The A-15 surface structure collapsed into bcc Nb when it was depleted of Al. This is perhaps not surprising but noteworthy.

4. The surface structure was disordered but remained crystalline. This is in contrast to the behavior observed in semiconductors where much smaller doses produce truly amorphous layers. The materials probably underwent a certain amount of self healing during implantation. This is more likely to occur in materials with metallic bonding than in those with covalent bonding. In fact, it is extremely difficult to render a metal amorphous.

5. The implanted layer which was originally disordered bcc Nb transformed into A-15 at 800°C and remained so as high as 980°C. We believe this was due to epitaxial recrystallization[1]. Soeder and Stritzker[9] have made A-15 Nb_3Sn and Nb_3G3 by implanting Sn and Ge into thin Nb foils. However, they were unable to stabilize A-15 Nb_3Si by the same method. This suggests the importance of epitaxy in our case.

6. The transition temperature of the implanted layer (5.6 K) was much lower than one would have expected from the empirical predictions. This is nevertheless higher than the T_c of the Ti_3P form of Nb_3Si which is 0.3 K[10]. The question is whether the T_c is low because A-15 Nb_3Si inherently has a low T_c or whether it is because it was not made with the proper lattice parameter and long range order. Typically, T_c increases with decreases in lattice parameter and increases in L.R.O. One might be tempted to believe that the disorder produced by the high dose implantations would degrade T_c. However, many experiments have been performed recently on irradiation damage in A-15's [11,12,13]. Although T_c is indeed degraded by high doses, a material invariably retrieves its initial T_c upon subsequent annealing. The author, therefore, believes that implantation induced disorder cannot be the cause of the low T_c observed. The ordering anneals did not seem to significantly affect T_c. It is to be expected that after the recrystallization anneals at ~900°C, there would be a certain amount of anti-site disorder; Al and Si atoms would have exchanged their positions with the Nb atoms. However, contrary to the behavior observed in Nb_3Al and $Nb_3Al_{0.9}Si_{0.1}$, the ordering anneals do not seem to have enhanced T_c;

one would therefore expect that the anti-site disorder has remained.

CONCLUSION

In conclusion, ion implantation has been used successfully to create a metastable phase of significantly altered composition. The starting material was A-15 $Nb_3Al_{0.9}Si_{0.1}$. The surface layer was depleted of Al by a diffusion anneal. Si was then implanted into the material with doses as high as 20 At.%, creating a disorderd region. By subsequent annealing the implanted layer recrystallized epitaxially on the substrate into A-15 $Nb_3Al_{0.2}Si_{0.8}$. Possible explanations for the low T_c in our case are being off stoichiometry, poor long range order, strain effects due to the epitaxial growth, or persistant lattice defects in the A-15 layer.

ACKNOWLEDGMENTS

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TABLE I

DOPANT PROFILE CHARACTERISTICS FOR Si^+ into Nb_3Al

ION ENERGY E KeV	RANGE R_p A	RANGE STRAGGLING R_p A
50	358	199
70	495	263
100	707	353
120	851	409
150	1069	490
170	1216	541
200	1438	615

DOPANT PROFILE CHARACTERISTICS FOR Si^+ into Nb

E	R_p	R_p
50	341	0
70	470	270
100	667	367
120	801	423
150	1003	509
170	1140	563
200	1345	639

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FIGURE CAPTIONS

Figure 1. R.E.D. photo of A-15 $Nb_3Al_{0.9}Si_{0.1}$ (80 KeV).

Figure 2. Indexing of the diffraction rings of Figure 1.

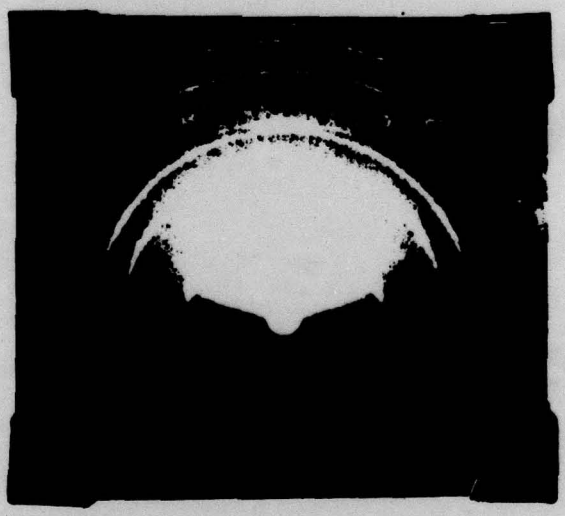
Figure 3. R.E.D. photo of the $Nb_3Al_{0.9}Si_{0.1}$ surface after the diffusion anneal and the Si implantation (80 KeV); the structure is disordered bcc Nb.

Figure 4. Indexing of the diffraction rings of Figure 3.

Figure 5. A comparison of the diffraction lines for the Ti_3P and A-15 structures.

Figure 6. Superconducting transition of the Si implanted layer.

Figure 7. Superconducting transition as a function of magnetic field.



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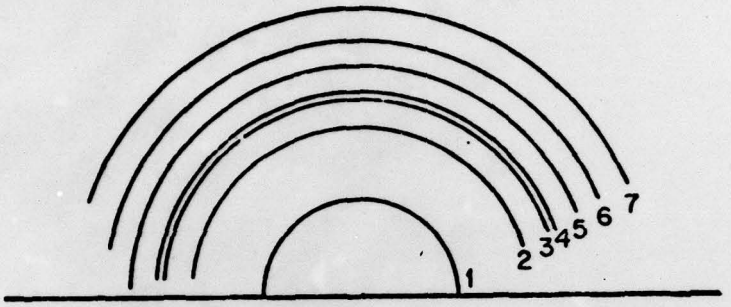

1{210}, 2{321}, 3{421}, 4{332}

5{440}, 6{610}, 7{622}

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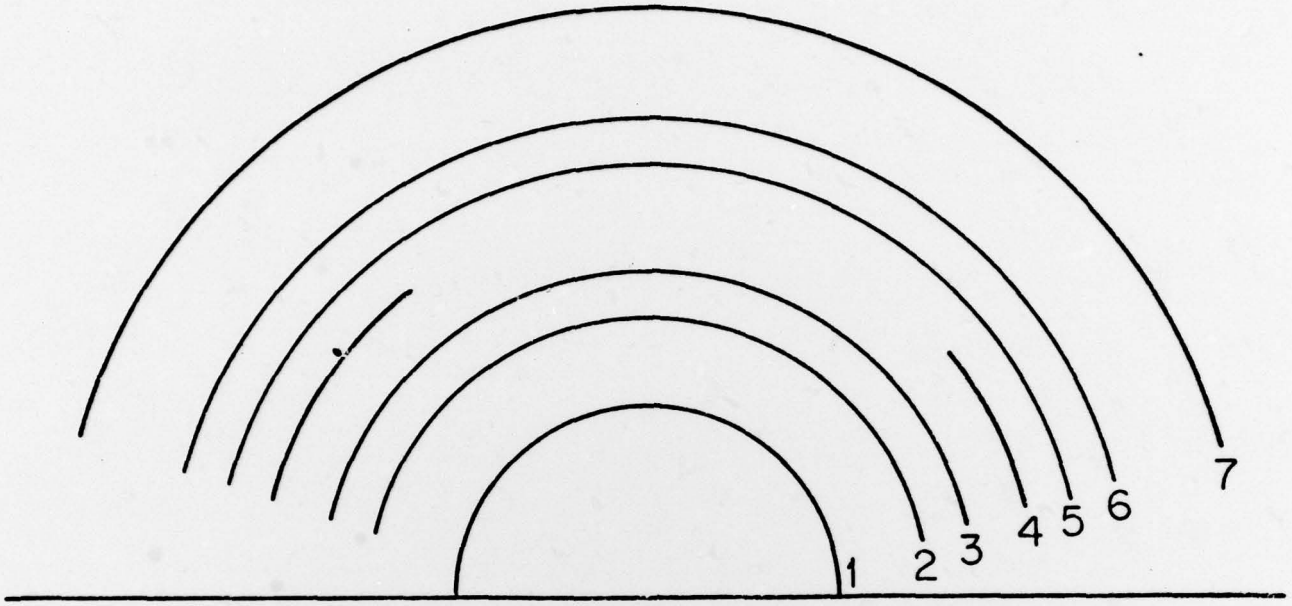
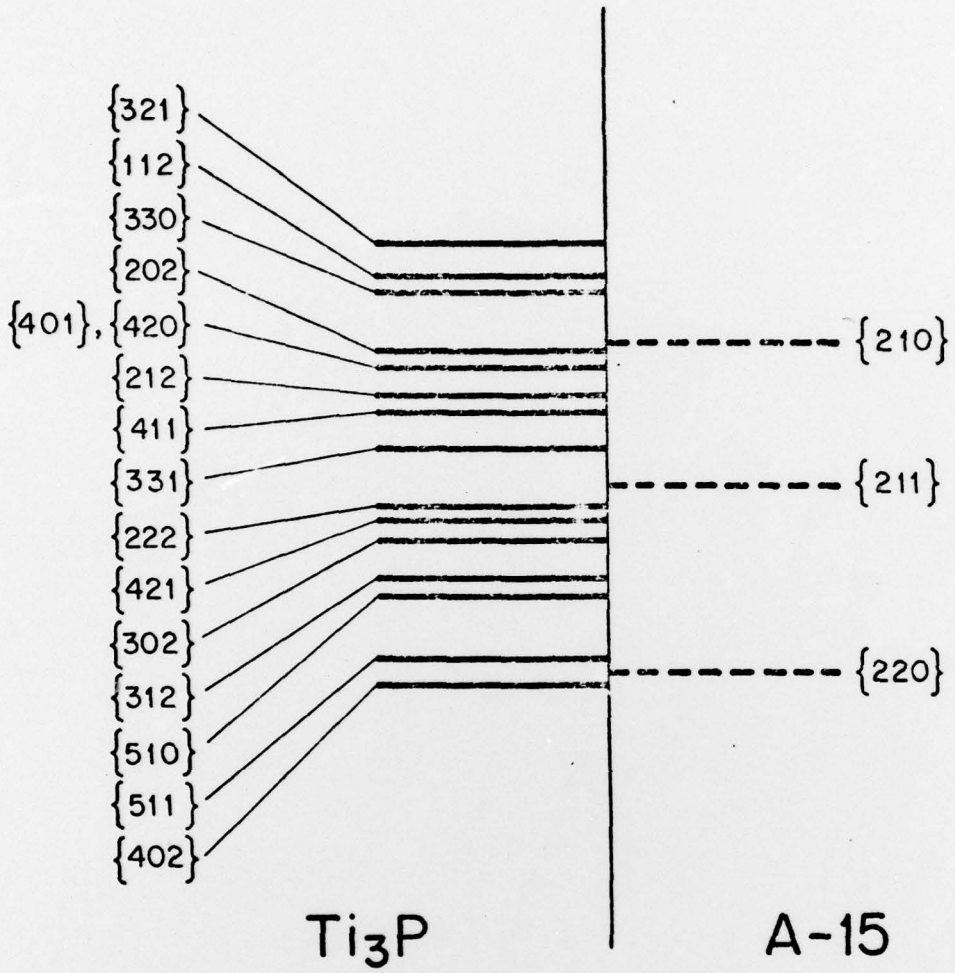


1{110}, 2{100}, 3{211}, 4{220}

5{310}, 6{222}, 7{330}

Figure 4. Indexing of the diffraction rings of Figure 3.



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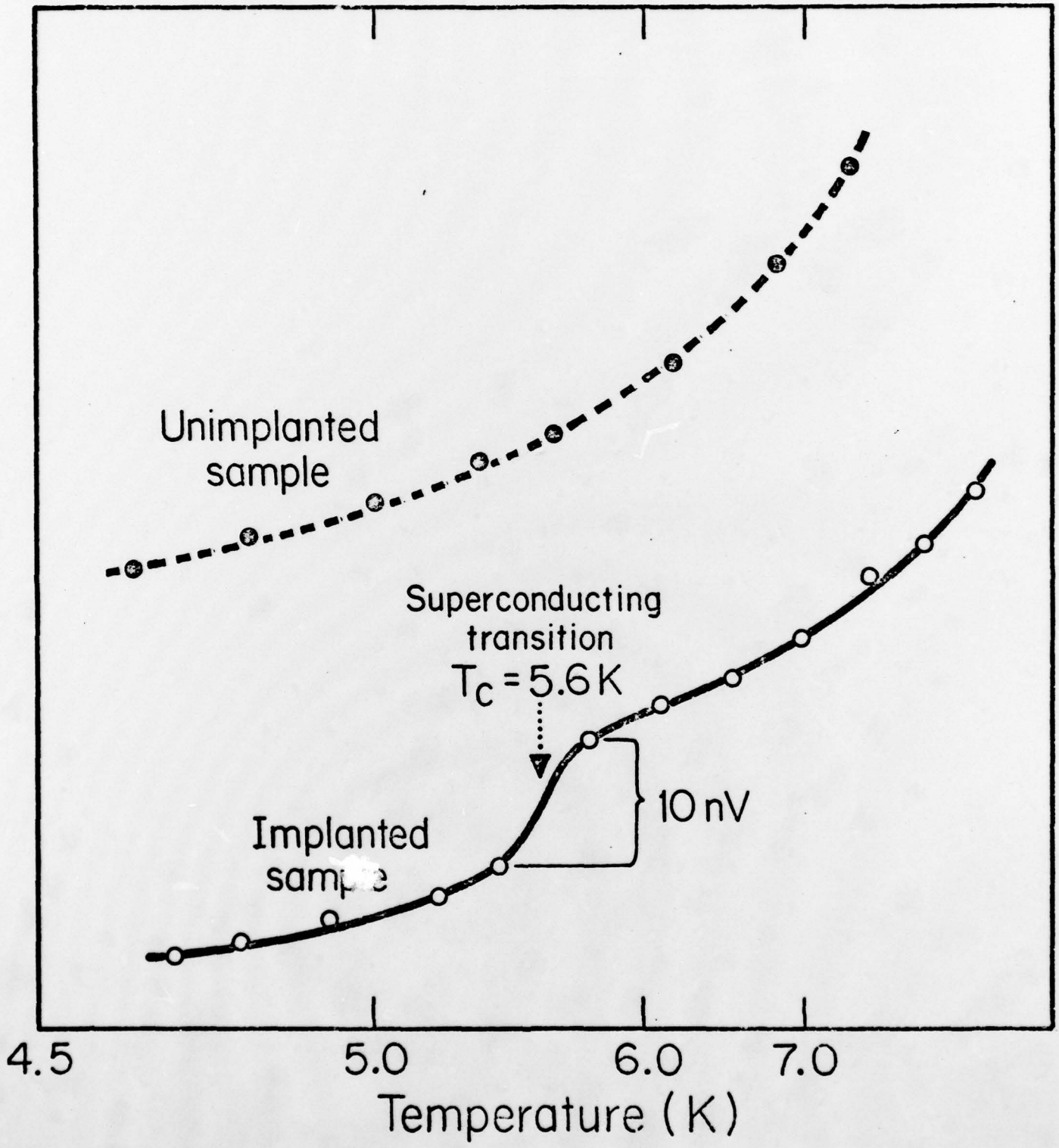

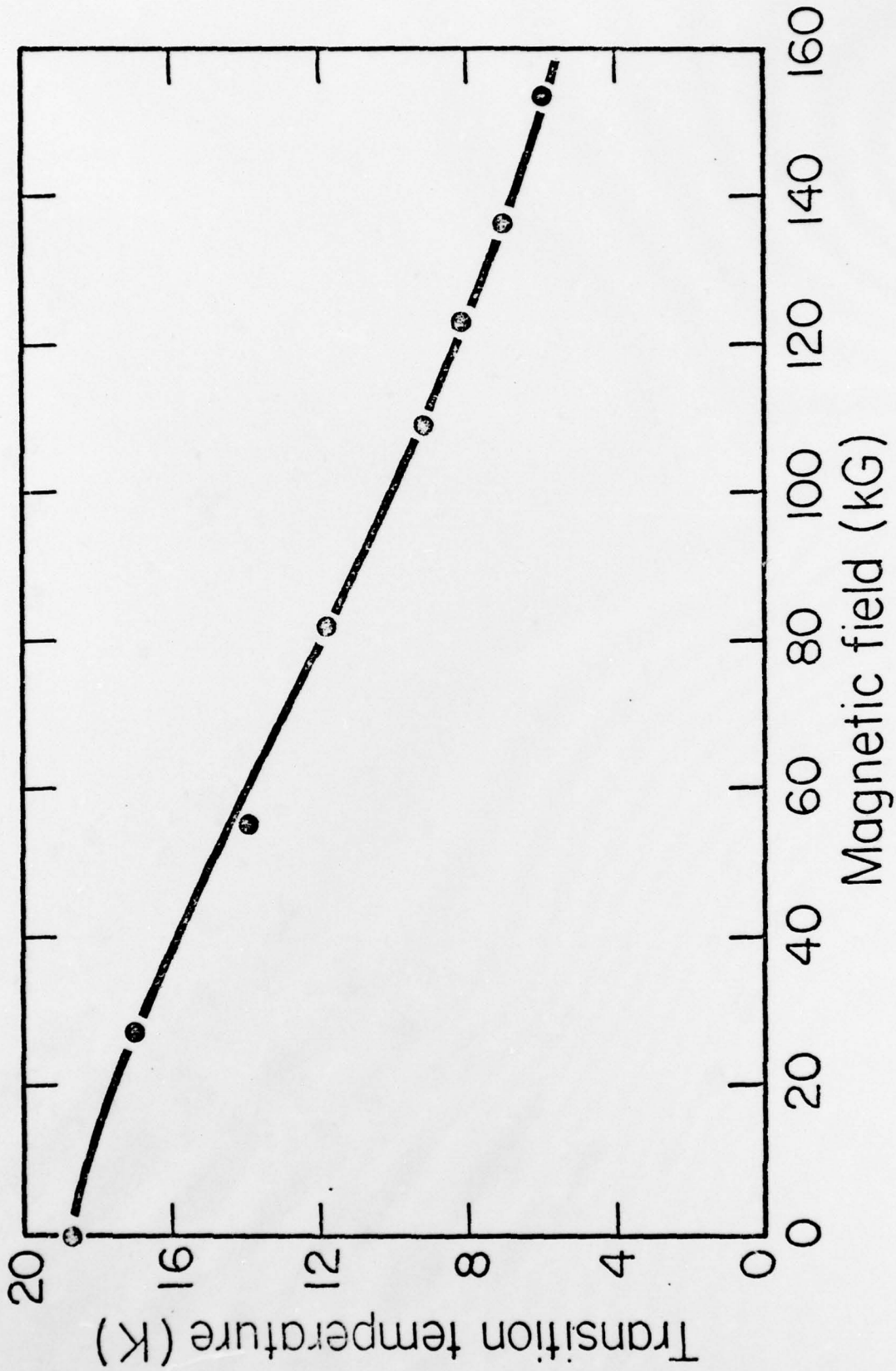

Figure 7. Superconducting transition as a function of magnetic field.